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DEVELOPMENT OF HEAT-RESISTANT Mg-Be ALLOYS
AS A CLADDING MATERIAL FOR FUEL ELEMENTS

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Mg-Be alloys are recognized as valuable materials for gas-cooled reactors. The idea of increasing heat-resistance of magnesium alloys by addition of beryllium in quantities considerably exceeding the limit of solid solubility of beryllium in magnesium and the mechanism of the protection effect of beryllium on the magnesium oxidation were considered in the previous works /1, 2/.

In the report presented the results of investigations of the advanced technology of preparation of Mg-Be alloys, containing considerable quantities of beryllium, as well as some new data about properties of these alloys are discussed.

I. TECHNOLOGY OF PREPARATION OF Mg-Be ALLOYS

The need of Mg-Be alloys of high purity to be used as a thin-layer protective claddings for fuel elements makes the addition of beryllium into magnesium from salts, for example, Na_2BeF_4 , BeCl_2 etc. practically unacceptable.

The only method which permits us to minimize the flux impurities in the ingots with a simultaneous provision of a considerable beryllium concentration in alloys is an addition of beryllium from double or triple beryllium alloys with some other metal. Processing magnesium fusion under the protection of special covering fluxes in open crucibles and the alloyage are effected at the temperature of 750-850°C. In this case the maximum Be concentration in the ingots did not exceed 0.03-0.04 % weight (MB-3 alloy).

It is possible to increase the beryllium content in the ingots up to 0.07-0.09 by increasing the temperature of an alloy up to 900-950°C, but in this case the quantity of impurities in ingots increases also because of flux and oxide inclusions. The preparation of alloys in hermetically closed crucibles permits to melt the alloys with no covering fluxes.

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In this case beryllium can be added into magnesium at the temperatures near the boiling point of magnesium. After melting and a subsequent rapid cooling of the alloy to the temperature of casting, the alloy pouring takes place in the air without any protective fluxes. The optimum technology of the preparation of the alloy in closed crucibles consists in melting of an alloy at 1000-1050°C and casting it at 720-750°C with minimum time-delay at these temperatures.

Thus the ingots of 15-20 kg containing 0.09-0.20 % weight of beryllium can be obtained (MB-4 alloy) /3/ .

Mg-Be alloys containing aluminium, calcium, silicon and thorium as a third component were obtained by means of this method. Investigations of effectiveness of introducing beryllium into liquid magnesium from different alloys showed that the double Si-Be alloy is most acceptable in this case.

Mg-Si-Be alloys thus obtained have a sufficient homogeneity of the structure and stable grain-size at high temperatures (Fig. I). Composition and some physical properties of alloys are given in tables I and II.

The method of powder metallurgy is one of the most economical methods of preparing the magnesium alloys with a higher beryllium content. We have prepared heat-resistant powder Mg-Be alloys with 2-10% and more percent of Be content (MMb alloys) /3, 4/.

Magnesium powder of the 160-50 microns grain-size with a surface oxidation less than 0.18-20% weight were used as initial materials. The total content of impurities (without oxygen) was not more than 0.2% weight.

The grain size of beryllium powder was 50, microns, oxidation of the surface was 1.5-2% weight and general purity was better than 99.5%. The technological scheme commonly used consists of subsequent operations of the formation and dry mixture of the charge, of cold briquetting and hot extruding of the powder. The porosity of the specimens from Mg-Be alloys obtained by hot extrusion at the optimum conditions is practically zero. Fig. 2 shows some specimens prepared of Mg-Be alloys. The composition and some properties of the alloys are given in tables I and II.

TABLE ICOMPOSITION OF ALLOYS

Composition of alloys % weight	Be	Si	Al	Th	Fe	Mn	Ni	Cu	MgO	Ca	Zr
Type of alloy											
ПМБ	from 0.5 to 32	-	0.01	-	0.04	-	0.001	0.005	0.2 0.3	-	-
МБ-3	0.04	0.5	-	-	0.01	0.001	0.001	0.005	-	-	-
МБ-4	0.08- 0.15	0.7	-	-	0.01	0.001	0.001	0.005	-	-	-
Mg-Al- Fe	0.04	-	0.5	-	0.01	0.001	0.001	0.005	-	-	-
Mg-Th- Be	0.04	-	-	3	0.01	0.001	0.001	0.005	-	-	-
Mg-Ca-Zr-Be	0.04	-	-	-	0.01	0.001	0.001	0.005	-	0.5	0.5

2. MECHANICAL PROPERTIES OF Mg-Be ALLOYS

Some properties of Mg-Si-Be alloys МБ -3, МБ-4 and powder alloys ПМБ -2 and ПМБ -5 are given in table II.

The physical properties of the alloys are almost the same as the properties of pure magnesium while the mechanical properties are different.

The mechanical properties of powder Mg-Be alloys in main part depend upon the quantities of fine-dispersed magnesium oxide on the surface of particles of the magnesium powder. The fine-dispersed oxide phase in the magnesium matrix results in the increase of the hardening and strengthness of alloys with the increase of the testing temperature.

The addition of the fine-dispersed beryllium phase into the magnesium metal-ceramic alloy results in the alloy additional strength although it is accompanied by some decrease of plasticity of alloys /4/.

The minimum plasticity of alloys at decreased rate of deformation is observed at 300-350°C (Fig. 3). The plasticity of alloys increases with the increase of the testing temperature, but plasticity of magnesium metal-ceramic that does not contain beryllium gradually decreases. This is evidently connected with the increase of the plasticity of a magnesium matrix which in case of HME alloys consists of a saturated solid solution of beryllium in magnesium.

It is necessary to note that the use of the methods of the powder metallurgy for preparing um Mg-Be alloys makes it possible to introduce easily the fine-dispersed magnesium oxide into the alloys and to improve their strength at the temperatures much higher the permissible operating temperatures of usual alloys on magnesium base.

3. OXIDATION OF Mg-Be ALLOYS

The results of the tests of powder alloys in the air and in technical carbon dioxide (0.1-0.2% H₂O) under pressure of 50 atm. showed that the Mg-Be alloys are corrosion resistant at the temperature of 580°C for more than 5000 hours if the beryllium content in alloys is equal to 2% or more (Fig. 4). The qualitative tests of corrosion-resistance of HME alloys were performed under these conditions during 12000 hours (fig. 5).

The electronographic and electronmicroscopic studies of the oxide films of HME alloys showed that their structure and phase composition as well as those in the case of distilled alloys depend upon beryllium content in the alloy, type of aggressive agent, temperature and period of oxidation /1, 2/. The essential feature of oxide film structure of HME alloys as well as of oxide films of distilled alloys is their double-layer structure which is especially distinct when the alloys are oxidized in the carbon dioxide.

The rate of oxidation of Mg-Si-Be alloys in the air and in the carbon dioxide (Fig. 4) slightly differs from the rate of oxidation of double Mg-Be alloys with the same beryllium content /2/. The alloys containing 0.08-0.15% of beryllium are not destroyed being tested in the air and in the carbon dioxide at 520°C for a period of more than 3000 hours. The resistance of these alloys is higher than that of alloys of

TABLE II

SOME PHYSICAL AND MECHANICAL PROPERTIES OF MAGNESIUM AND MAG-
NESIUM- BERYLLIUM ALLOYS

Properties	Physical properties				Mechanical properties													
	density gr/cm ³ at 20°C	t°C of melting	Spe- cific elec- tric con- ducti- vity /Ω.cm	Coef- ficient of li- near expan- sion in the in- terval of 20°C x 10 ⁻⁶ 1/deg.	20°C	20°C	20°C	200°C	200°C	200°C	200°C	200°C	200°C	200°C	200°C	200°C	200°C	200°C
Alloys					kg/sq.mm	kg/sq.mm	%	kg/sq.mm	kg/sq.mm	kg/sq.mm	kg/sq.mm	kg/sq.mm	kg/sq.mm	kg/sq.mm	kg/sq.mm	kg/sq.mm	kg/sq.mm	kg/sq.mm
Magnesium	1.738	650 ⁺¹	4.70	25.8	18	9	9	34	5.4	2.3	46	1.8	1.4	52	0.8	0.4	60	0.4 - 78
ME -4	1.746	648 ⁺²	4.40	23.7	22	15	6	38	8.5	5	29	3.8	1.9	48	2	0.9	64	0.9 - 82
ME -3	1.742	648 ⁺²	4.40	23.6	20	13	8	36	7	4	36	2.9	1.7	58	1.3	0.7	69	0.8 - 90
ΠME -2	1.741	649 ^{+1.5}	4.85	23.0	26	19	5.2	40.5	12	7	14	8	4.8	16	5	3	27	2.8 - 49
ΠME -5	1.743	649 ^{+1.5}	4.90	22.8	24	19	5	40.5	11.5	7.9	12	7.5	5	15	5	3.1	26	3 - 49

Note: The samples tested are prepared hot extruded rods after their thermal treatment were tested.

"magnox" type /5/. The resistance of Mg-Si-Be alloys decreases under pressure of 50 atm. of the carbon dioxide as compared with the alloy resistance under pressure of 1 atm. Nevertheless MB -4 alloys are not destroyed under these conditions for 5000 hours at 520°C or less (Fig. 4). MgO and small quantities of BeO are detected in oxidized films of y Mg-Si-Be alloys by electronographic methods.

The heat-resistance of the alloys that content Si, Al, Ca, Zr and Th depends first of all upon the beryllium content. Additions of Si, Al and Zr in quantities that are given in the Table I have no appreciable effect upon the heat-resistance of the alloys, but Th addition decreases the heat-resistance of the alloys.

Calcium additions slightly improve the heat-resistance of alloys in the air but deteriorate the same in carbon dioxide.

The protective properties of beryllium on oxidation of magnesium-beryllium alloys are characterized by predominant diffusion of beryllium to the surface of oxidation and formation of BeO layer in the lower layers of oxide films. This layer is low-permeable for beryllium ions and practically impermeable for Mg ions. The process of magnesium oxide reduction by beryllium plays an essential part in formation of a sublayer.

The increase of a content of BeO in oxidized films results in improvement of their strength and cohesion to the alloys.

Long time heat-resistance of Mg-Be alloys is determined by beryllium content despite the fact that alloys of small beryllium content (0.03-0.2%) show a lower rate of oxidation at the initial oxidation stage than the alloys that have a larger content of beryllium (Fig. 6). The value of concentration of Be which is necessary to provide a long time heat-resistance of alloys substantially depends upon the technology of the preparing the alloy its structure and a quantity of other alloying components in it. Thus the heat-resistance of powder alloys is determined by the dispersion of beryllium component, its distribution and is achieved at a considerably higher content of beryllium in an alloy than in alloys with high dispersed beryllium prepared by a distillation met-

hod.

Magnesium alloy corrosion resistance in different gases depends to the great extent upon the humidity of these media.

This report deals with the study of the behaviour of various Mg-Be alloys at the temperatures of 500-580°C in the air containing 10% of H₂O and in the carbon dioxide containing 6.5% of H₂O. Comparing the curves (Fig. in 7, 8) with those (Fig. 4, 6) as well as with the results given in the work /2/, one finds that the moisture has no appreciable effect upon the alloy oxidation rate at the first stage of oxidation. However destruction of Mg-Be alloys prepared by distillation with small amounts of beryllium begins after some time of oxidation while at the same time only porosity and swellings appear in some places of the alloys with a some higher beryllium content. The porosity that appear under the same conditions of oxidation in Mg-Be alloys prepared by powder metallurgy is much smaller.

The alloys are corrosion-resistant for a long time in damp gases and in the water vapour at 520°C.

Chemical analysis of samples of the alloys tested previously in the damp medium showed that the process of hydrogenation of the alloys takes place during oxidation (Table III).

TABLE III
HYDROGEN CONTENT (CM³/100 G OF METAL) IN MAGNESIUM
AND Mg-Be ALLOYS

Magnesium or Mg-Be alloy	After oxidation in	
	Before testing	the air (10% H ₂ O) for 750 hours, 580°C
Magnesium Mg-I	9	The samples were destroyed
Distilled magnesium (druse)	13	"
Distilled alloy (0.27% of Be)	16	86
IME -5 alloy	20	80

The harmful effect of water vapour upon Mg-Be alloys

during oxidation in a damp air and carbon dioxide is probably related to penetration of hydrogen ions formed during the interaction between the water vapour and Mg and Be into the lattice of the magnesium oxide and to its diffusion to the oxide-metal boundary and into the metal /6, 8/.

The atomic hydrogen dissolved in an oxide film and in a metal may reach the equilibrium with molecular hydrogen in pores, cracks or in other defects of the oxide film and the metal since the equilibrium pressure of atomic hydrogen at considered temperatures and the molecular hydrogen pressure of 1 atm is equal to 10^{-12} atm, the pressure of molecular hydrogen in this case may reach a high value and destroy the material.

A favourable effect of beryllium on magnesium oxidation in damp gas media is probably related to strengthening of oxide films and their better cohesion to the metal and the decrease of hydrogen penetration into them.

Therefore in case of Mg-Be alloys the atomic hydrogen diffuses into the alloy in considerable quantities without destroying oxide films and appears in molecular state on the defects of an alloy, and this results sometimes in swelling of the alloy. In case of oxidation of pure magnesium and Mg-Be alloys with a small beryllium content ($\sim 0.005\%$), the metal-oxide film boundary or the film itself is the weakest point of the system. These materials are oxidized with no formation of protective films. A smaller effect of hydrogen in powder Mg-Be alloys may be due to strengthening of these alloys by particles of beryllium MgBe_{13} intermetallide and oxide films.

Ignition temperature of pure magnesium in the air is below its melting point and equals to $632 \pm 2.5^\circ\text{C}$; and in pressurized carbon dioxide it is equal to $650 \pm 750^\circ\text{C}$ (Table IV).

Ignition temperature of Mg-Be alloys both in the air and the carbon dioxide rises considerably with the increase of beryllium concentration.

TABLE IV

IGNITION TEMPERATURE OF ALLOYS

Alloy	Content of beryllium in alloy (%)	Ignition temperature		
		air	CO ₂	60 atm
Pure Mg	-	632 [±] 2.5		650-750
Mg-Si-Be	0.09-0.15	650 [±] 3		720-750 ^{x)}
ПМС -2	2	665 [±] 5		770-850 ^{x)}
ПМС -5	5	700 [±] 15		"-
Mg-Be (distill.)	0.5-2	"-		"-

x)

Temperature at the beginning of intensive oxidation.

Heating up to high temperature (above 700°) in the pressurized commercial carbon dioxide results in the alloy destruction but the total inflammation does not take place.

5. COMPATIBILITY OF Mg-Be ALLOYS WITH DIFFERENT MATERIALS

Mg-Be alloys are incompatible with Al, Zn, Cu, Ni at temperatures of 450-500°C since formation of eutectics and intermediate layers of low corrosion-resistance results in the alloy destruction. However if the beryllium content in alloys reached 2-5% weight the alloys resisted to a considerable overheating above the temperature of an eutectic formation without destruction. The Mg-Be alloys are compatible with chromium, titanium, carbon and uranium for long periods at the temperature of 580-600°C (Table Y).

Mg-Be compatibility with Zr, Zr-Cu alloys, stainless and low carbon steels depends upon the method of fabrication of Mg-Be alloys, upon oxidation level of contact surface, of tested materials and also upon the pressing force between the samples (Table Y). Preliminary oxidation of the contact surfaces of the materials makes them quite compatible at temperatures in the range of 500-550°C.

TABLE V

Tested material	t°C of test	Time of test (hrs)	Alloys	Degree of diffusion, interaction	Note
Zr, Zr-Cu	450	1650	Mg-Si-Be, ПМС-5	Interaction was not observed	Initial pressing force 15 - 20 kg/mm ²
	520	650	Mg-Si, Be- Mg-Be	Diffusion layer up to 300 m	Initial pressing force 15 - 20 kg/mm ²
		1000	ПМС -5	Diffusion layer 15 - 20 m.	
	450		Mg-Be, Mg-Si-Be	Diffusion layers up to 400 m	Corrosion destruction in contact place
(0.5-1% Cu)	580	650	ПМС -5	"-	Tablets of Zr-Cu alloy were previously oxidized
		1000	ПМС -5	Interaction was not observed	
Steel 1x18H9T	450	1650	Mg-Be, Mg-Si-Be	" - "	
		200	" - "	" - "	Initial pressing force 1 kg/mm ²
	520	200	" - "	Diffusion layers 0.5-1 mm	Initial pressing force 15 - 20 kg/mm ²
Steel 3	580	120	" - "	Interaction was not observed	Initial pressing force 1 kg/mm ²
		200	" - "	Diffusion layers 1 - 2 mm	Initial pressing force 15-20 kg/mm ² . Diffusion of Ni, Fe into Mg alloys

Tested material	t°C of test	Time of test (hr)	Alloys	Degree of diffusion, interaction	Note
Ti, Cr, C	450	1650	Mg-Be, Mg-Si-Be	Interaction was not observed	Sintering of tablets Ti and Cr with Mg-Be alloys takes place
	580	900	"-"	"-"	
U	520	1000	Mg-Be	Poor diffusion interaction with formation UBe_{13} , solution of U in Mg	Impoverishment of Mg-Be alloy in beryllium is observed

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APPLICATION OF Mg-Be ALLOYS FOR COATING OF ROD
FUEL ELEMENTS

Tubes were extruded from MB and ПМБ alloys and used afterwards for coating of uranium rods of fuel elements of the heavy water has reactor (I, 9/. The fuel elements of 5 mm outer diameter and 4000 mm long were coated with a layer of Mg-Be alloy that was 0.5 mm thick. Rod fuel elements, were tested for a long time in the air and in the static carbon dioxide at the pressure of 50 atm (Table VI, Fig.9).

TABLE VI

Test conditions Coating material	Air 1atm.		Commercial Carbon dioxide 50 atm. (humidity) 0.1% weight)	
	520°C	470°C	520°C	550°C
Mg-Si-Be alloy				
MB -4	3000 hr ^{xx}	11000 hr ^x	5000 hr ^x	2000 hr ^{xx}
Powder alloy ПМБ -2 -		11000 hr ^x	11000 hr ^x	7000 hr ^{xx}
"- ПМБ -5	2000 hr ^x	11000 hr	11000 hr ^x	11000 hr ^x

^x

Tests are continued without coating destruction.

^{xx}

Time interval before coating destruction.

It is clear from the data given in Table VI that powder alloy ПМБ -5 is the most corrosion-resistant material compared with all other materials having been tested.

As the experiments showed fuel elements coated with the alloy endure overheating in the air and also in the carbon dioxide without destruction up to the temperature of 650°C.

A decrease of the beryllium content in alloys during long-time tests is observed due to burning out beryllium during oxidation of alloys as well as to beryllium diffusion into uranium and formation of intermetallide compound UBe₁₃. The thickness of the impoverished layer in distilled alloys which have a high dispersed beryllium component reaches 50 μ on the boundary alloy-carbon dioxide and 100 μ on the boundary alloy-uranium interface (Fig.10). It was found out after

testing for 1500 hours at 580°C. The impoverished layer in both cases equals to 30-40 μ after testing for 2500 hours at 520°C.

The decrease of beryllium content is less observed in powder alloys than in distilled alloys. It is explained first of all by lower dispersion of beryllium in alloys. Tests of fuel elements proved that the increase of roughness of the protective coatings under static conditions in the air and carbon dioxide at 550°C, after 7000-10000 hours does not exceed 15-20 micron and is mainly due to corrosion processes on the coating surface.

Tensile tests of rod fuel elements showed that providing the maximum cohesion of coating to an uranium core one may considerably improve the plasticity of thin sheets of magnesium-beryllium layer. In this case the permissible elongation of coating, which is not followed by any breakdown in its air-tightness, may reach 15-20% (Table VII) even at a low rate of elongation.

TABLE VII

ULTIMATE DEFORMATION OF ПМБ -5 ALLOY COATING

Temperature of testing	250°C	300°C	400°C	500°C
Ultimate deformation of ПМБ -5 alloy coating, 0.5 mm thick	10-12%	16-17%	18-20%	20-25%

Note: A deformation rate under the test was equal to $7 \cdot 10^{-3}$ mm/min. The thermo-cyclic tests of the fuel elements were carried out in order to determine the strength of cohesion of the the coating with uranium, the plasticity and corrosion resistance of the coating. The tests were performed at temperatures 50-500°C. It was proved that protective layers of МБ -4, ПМБ -2, ПМБ -5 alloys stand 600-700 cycles without breakdown of the hermeticity of the coating and without exfoliation of the coating from the uranium core. The roughness rises in this case up to 150-170 μ and that is explained primarily by different coefficients of linear expansion of uranium and magnesium-beryllium alloys.

The radiation tests of the fuel elements in the reactor in the pressurized flow of carbon dioxide of 60 atm in the range of temperatures 150-580°C and in the integral neutron flux of 2.10^{20} n/sq.cm for 6000 hours showed that the protective coatings fabricated from the developed magnesium-beryllium alloys met the requirements of the coating material used under these conditions.

No corrosion or mechanical destructions of the fuel elements were observed, no failure in hermeticity. No changes were detected in the alloy structure because of irradiation.

CONCLUSION

Heat-resistant Mg-Be alloys containing the amounts of beryllium exceeding the limit of solubility of beryllium are developed. Investigations of the properties of these alloys as a cladding material for fuel elements (long-time heat-resistance, mechanical properties, ignition resistance, compatibility with other materials, leak-tightness of fuel elements coating under thermo-cyclic and reactor conditions) were performed.

The Mg-Be alloys be used as a cladding material for fuel elements of reactors cooled with carbon dioxide at the coating surface temperature of 520-530°C.

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Fig. 1. Structure of hot-pressed rods of Mg-Be alloys:
 a) МБ-4 alloy (x200); b) ПМБ -2 alloy (x340).

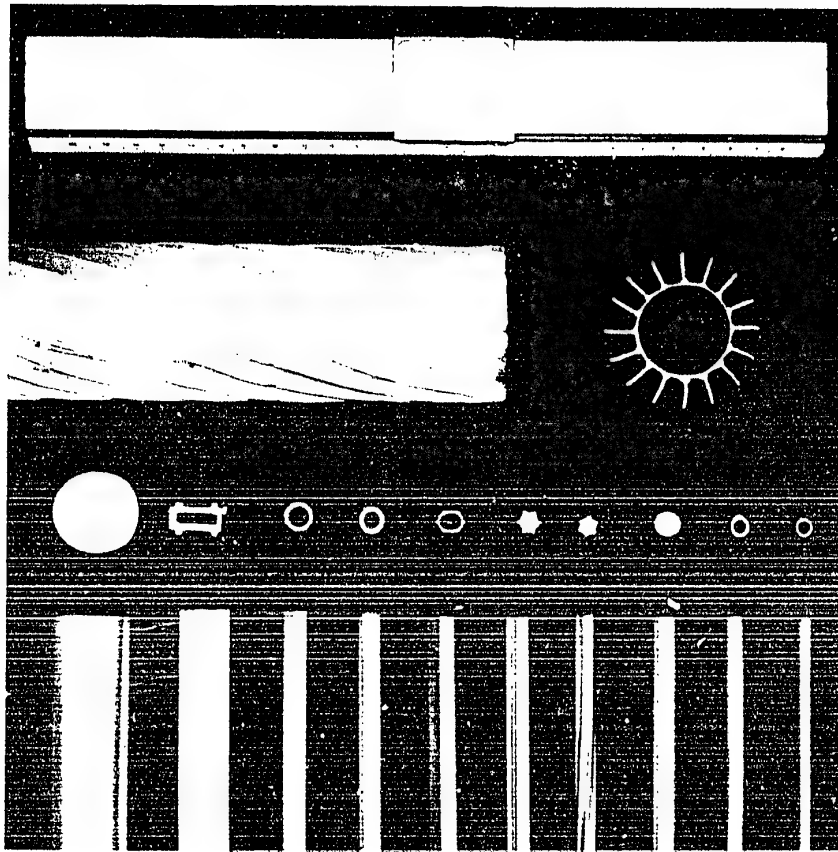


Fig. 2. Outward view of articles made of Mg-Be alloys.

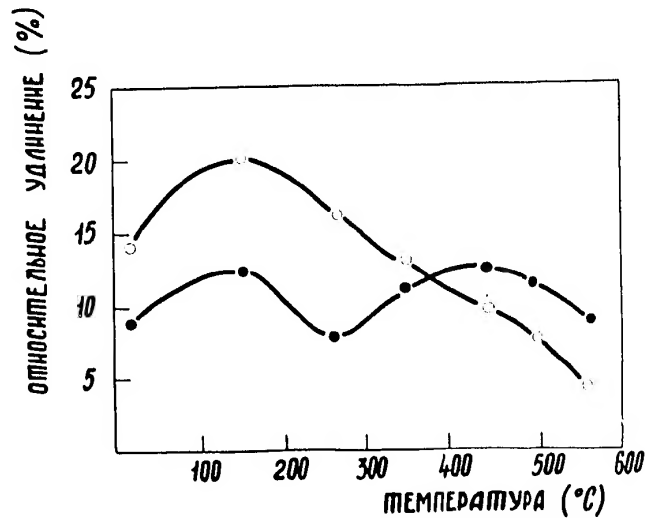


Fig. 3. Relative elongation of the powder magnesium alloys tested under low rated of tension (1.6% p/h)

○ - Mg + 0.3% MgO alloy

● - ПМБ-5 (Mg + 0.3% MgO + 5% Be).

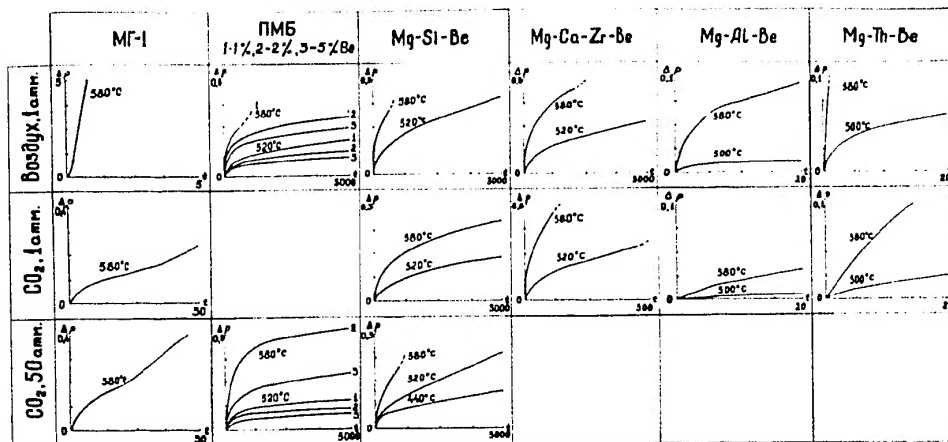


Fig. 4. Curves of oxidation of magnesium-beryllium alloys in different gases (ΔP - increase in weight mg/sq.cm t - hours).

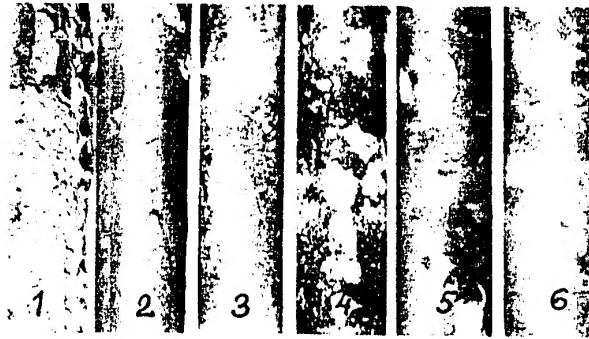


Fig. 5. Outward view of the samples after testing in the air at the temperature of 580°C :

- 1 - non-alloying magnesium, 0.5 hrs.;
- 2 - Mg-Si-Be alloy (MB -4), 400 hrs. (beginning of decay);
- 3 - distilled Mg-Be alloy; 0.5% Be, 1000 hrs;
- 4 - ПМБ -0.5 alloy, 500 hrs. (beginning of decay);
- 5 - ПМБ -2 alloy, 12000 hrs. (beginning of decay);
- 6 - ПМБ -5 alloy, 12000 hrs.

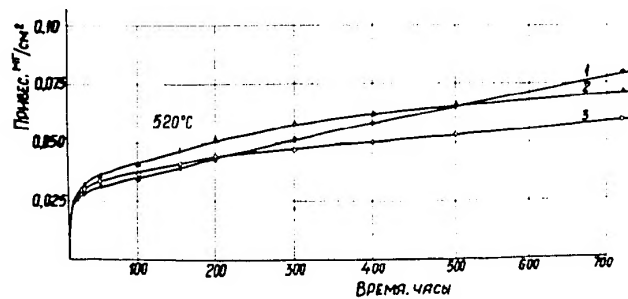


Fig. 6. Oxidation of Mg-Be alloys in the air:

- 1 - 0.16% ; 2 - 0.98% ; 3 - 2.69% Be.

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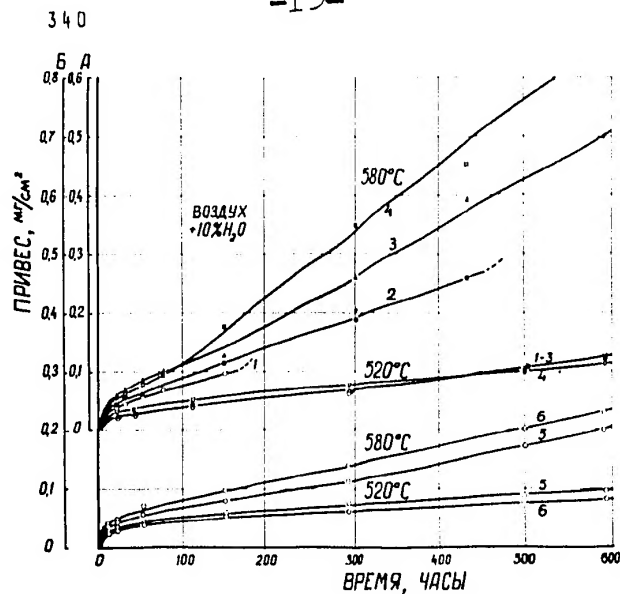


Fig. 7. Oxidation of Mg-Be alloys in damp air.

A - distilled Mg-Be alloys; 1 - 0.03%; 2 - 0.16%;
 3 - 0.27%; 4 - 0.7% Be
 B - ПМБ alloys; 5 - 2.5% Be; 6 - 5% Be.

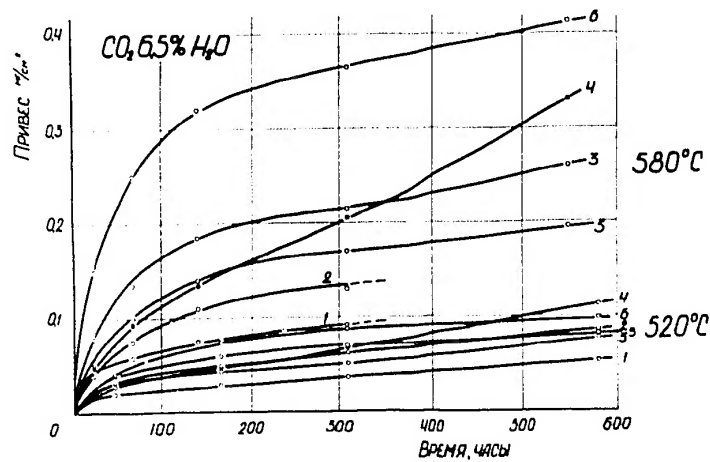


Fig. 8. Oxidation of Mg-Be alloys in damp carbon dioxide:
 1 - 0.03% Be, 2 - 0.16% Be; 3 - 0.7% Be (distilled alloys); 4 - 0.5% Be; 5 - 1% Be; 6 - 5% Be (ПМБ alloys).

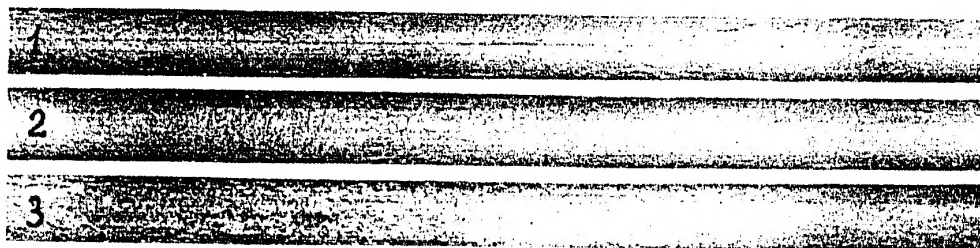


Fig. 9. The view of the surface of the fuel element samples before and after testing in the carbon dioxide at 50 atm:

- 1 - coating before testing;
- 2 - coating of ПМБ -2 alloy, 550°C 7000 hrs;
- 3 - coating of ПМБ -2 alloy, 550°C 7000 hrs;
- 4 - coating of ПМБ -5 alloy, 550°C 11000 hrs.
(x2.7).



Fig. 10. Impoverishment in beryllium of the coating made of distilled Mg-Be alloy, (1.32% Be) of the fuel element after testing in CO₂ at 580°C, 500 hrs.

- 1 - coating - gas limit;
- 2 - coating- uranium (x 200) limit.